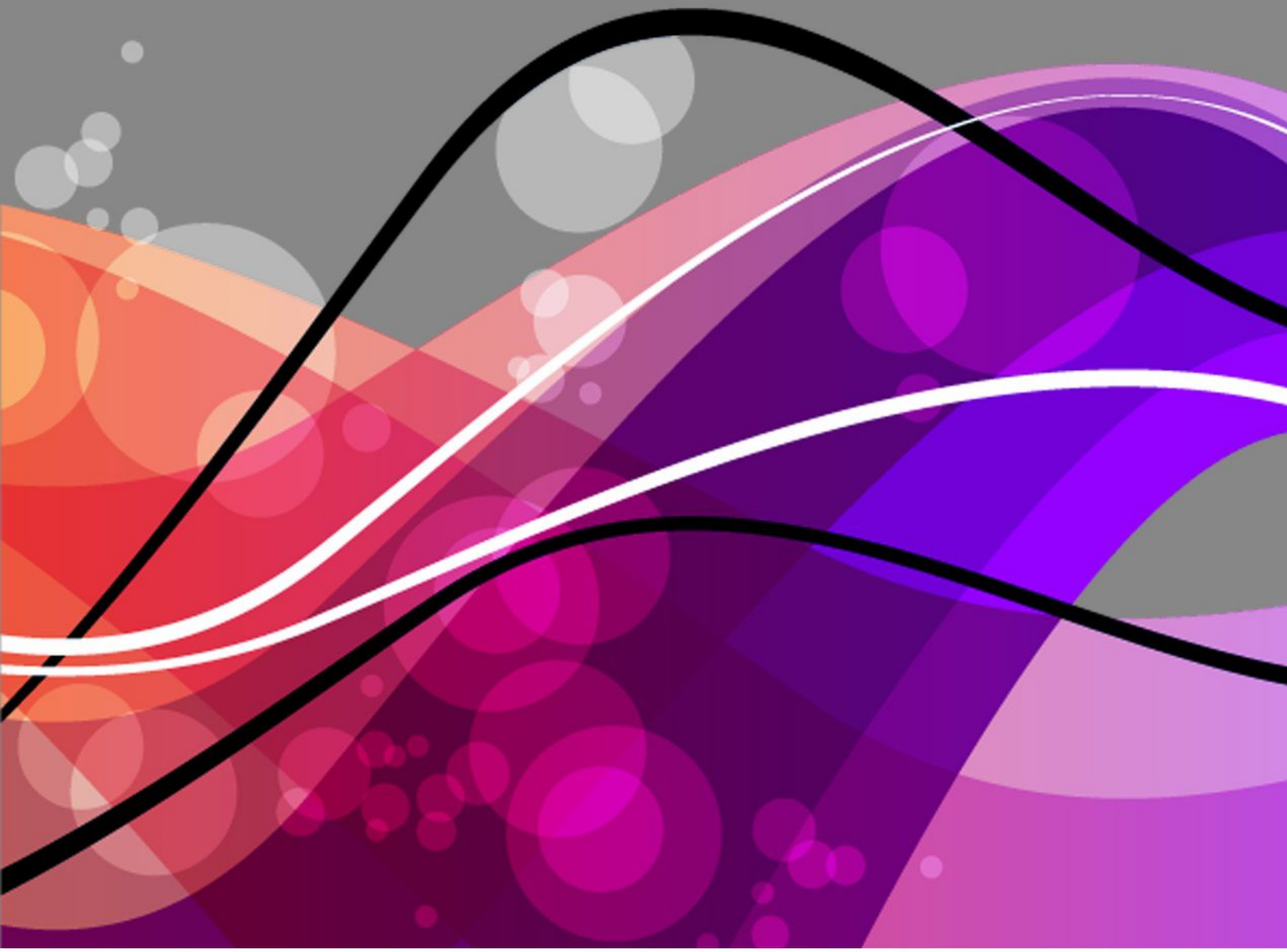
Introduction to Gas Chromatography by Lab-Training.com



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EXPERIENCE BEYOND BOOKS

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Author's Profile

Dr Saurabh Arora is a trained pharmacist with Master's and Doctorate degrees in pharmaceutics from reputed Indian institutes NPIER and Jamia Hamdard University respectively.

Managing Director

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He has setup 2 contract laboratories and a clinical research company along with managing and growing the existing business. His organization has grown multiple folds and he has been fortunate to spearhead the growth initiatives backed by a team of over 250 employees..

Specialties: Formulation Development, Analytical Development, Chromatography, Mass spectroscopy, GMP. GLP, GCP, Laboratory designing, Residue analysis, Project management, International business and all that goes into growing and managing a business

Founder

Lab-Training.Com

Lab-Training.Com is developing and offering a series of free and paid E-Learning courses on various analytical and laboratory techniques. He is responsible for the course concepts, course content creation and review and course execution.

Founder

Food Safety Helpline

Food Safety Helpline has been established to help Food Business Operators implement the Food Safety and Standards Act

Dr Deepak Bhanot is a seasoned professional having nearly 30 years expertise beginning from sales and product support of analytical instruments. After completing his graduation and post graduation from Delhi University and IIT Delhi he went on to Loughborough University of Technology, UK for doctorate research in analytical chemistry. His mission is to develop

training programs on analytical techniques and share his experiences with broad spectrum of users ranging from professionals engaged in analytical development and research as well as young enthusiasts fresh from academics who wish to embark upon a career in analytical industry.

Lab-Training.com

- Knowledge grows when shared with others. Our belief in this has contributed immensely towards growth of our web based portal for sharing our expertise and skills.
- Knowledge does not discriminate between national boundaries color of skin, religion, caste, gender and creed.

Our world class infrastructure, manpower skills and over 25 years of experience is now accessible to web based portal as we moved on from limited classroom training provider role over the last few years.

Our e-learning courses, articles and certificate programmes have been appreciated by industries, institutions, regulatory organizations and even individuals across the globe. There are constant demands for courses and articles on techniques of analytical interest and improvement of laboratory activities. We are bound to upgrade our content keeping the needs of our clients and followers in mind. It will be our endeavor to provide leadership in this key area of development.

Table of Contents

Introduction to GC Course and its objectives

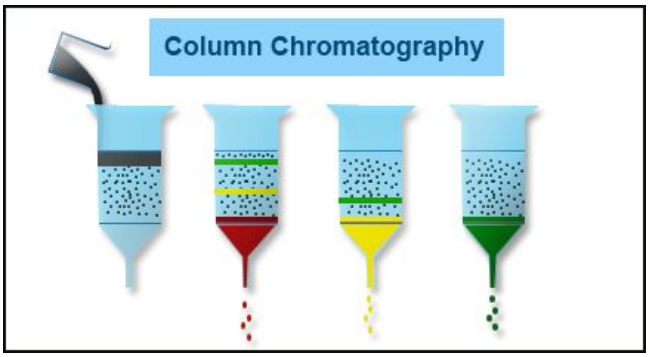
"It is possible to fly without motors, but not without knowledge and Skill"

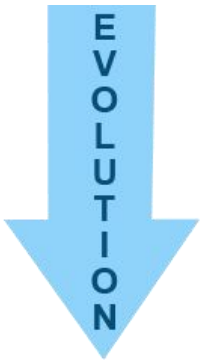
— Wilbur Wright

The overwhelming response to the HPLC e-book has encouraged us to move ahead with the other courses announced earlier. We understand that everyone has busy work schedules and today's hectic life style leaves you little or no time to refer voluminous books to learn any technique. However, for sustained growth learning has to be adopted as a lifelong habit. In an effort to make your learning task easy we embarked upon the e-learning course comprising of 10 Chapters which shall be forwarded to you in a phased manner. Each Chapter of 200-300 words length will provide functional aspects of GC and also present useful practical tips. Reading a Chapter and understanding it will not take more than about 10 minutes and you will get ample time to assimilate the content before you move to the next Chapter. The free programme is designed to give an insight into the technique and once your interest is captivated you can opt for full time advanced online or contact programmes. Such programmes will offer additional benefits of practical exposure and interaction with our technical experts.

Evolution of Gas Chromatography

"Tell me and I'll forget : show me and I may remember; involve me and I'll understand" --- Chinese Proverb





Chromatography originated in early 1900 when Russian Botanist Mikhail S. Tswett separated plant pigments using calcium carbonate packed glass columns. It was not until mid century that the technique was applied to develop paper chromatography, High Performance Liquid Chromatography and Gas Chromatography.

Chromatography helps isolate almost all components present in a mixture in pure form instead of just a single component starting with a small amount of the mixture (nano grams or microliter quantities). This was not possible earlier as the conventional methods were time consuming, required larger amount of sample and expensive solvents for isolation of a single component only. This important advantage contributed to the phenomenal growth and applications of different chromatographic techniques.

Martin and James in 1952 introduced Gas Chromatography in its present form and since its introduction it has become a premier technique of organic analytical separations with applications ranging from natural compounds, environmental studies, forensics and significantly in petroleum/ petro chemicals industry. GC permitted not only the rapid determination of already known components of complex mixtures but also revealed the presence of trace compounds that were not even known earlier.

Perkin Elmer introduced the first commercial Gas Chromatograph, model 154 Vapour Fractometer in the year 1955. Ever since growth of the techniques has resulted mainly due to advances in technology notably.

Injection Techniques

- Micro Syringes
- Packed column injectors
- Split/ Splitless capillary column injectors
- PTV
- Automated Liquid Injectors
- Pyrolysers
- Thermal Desorption
- Sampling Valves

Columns

- Packed Columns
- Fused Silica Capillary Columns

Detector

- Flame Ionization Detector
- Thermal Conductivity Detector
- Electron Capture Detector
- Mass Spectroscopic Detector
- Flame Photometric Detector
- Nitrogen Phosphorous Detector

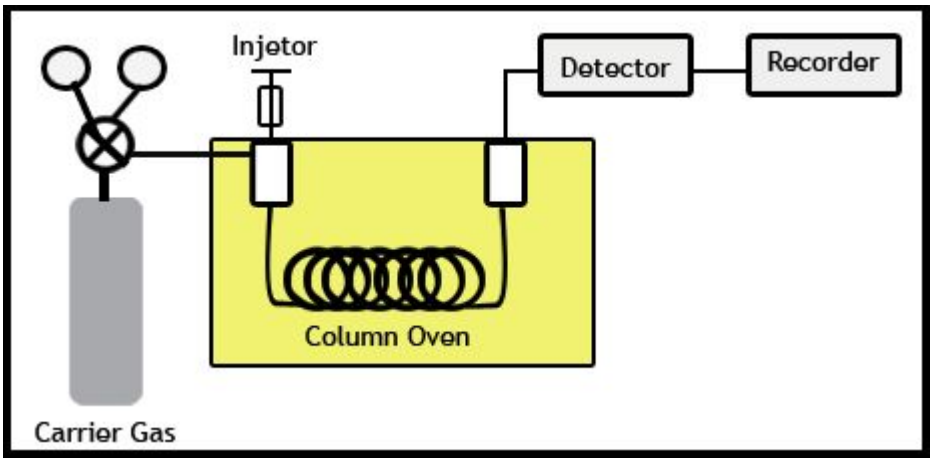
Special Application Configuration

- Natural Gas Analyzers
- Refineries Gas Analyzers
- Dissolved Gas Analyzers
- Simulated Distillation Analyzers
- Portable Gas Analyzers
- On line Gas Analyzers
- PONA/ PIONA Analyzers

The greatest impetus to the evolution of gas chromatography has been through introduction of hyphenated techniques such as GC-MS, GC-MS-MS, GC-HS, GC-FT-IR and GC-TGA-FT-IR which have further broadened the applications range of GC.

Introduction to Gas Chromatography and Its Parts

"The only source of knowledge is experience" --- Albert Einstein



The Gas Chromatograph appears to be a black box to the novice but an understanding of the components and their role will help raise your comfort level and help you generate data of highest reliability. You will also appreciate the role of preventive maintenance for long time usage and high reliability of results.

Gas Chromatography is helpful for separation, identification and quantification of component compounds in a mixture. In contrast to the High Performance Liquid Chromatography technique Gas Chromatography uses a gas as carrier mobile phase instead of a liquid. It is specially suited for compounds that are volatile, thermally stable and have low molecular weights.

The carrier gas is led at a constant flow rate to the column packed with the stationary phase. Before entering the column the sample mixture (gas or liquid) is injected into the carrier gas stream. The liquid mixture is vaporized to the gaseous state by the high temperature inside the injector before being led to the column. On reaching the column the sample components are selectively retained on the basis of physico – chemical interactions between the analyte molecules and the stationary phase. The mobile gas stream moving at a steady rate elutes the mixture components in a sequence determined by the operating conditions. Different detectors

are employed for detection and quantification of eluted compounds. You will now be introduced to the significant role of each part of the GC system.

Carrier gas

Carrier gas serves to transport the injected sample to the system. The gas selected for the purpose is inert to the sample and column packing material. Care should be taken to remove residual moisture or other gaseous impurities.

Injectors

Injectors are used to provide constant volume injection of sample into the carrier gas stream. Inertness and reproducibility of injection are essential to maintain high level of accuracy. The injectors should be able to handle small volumes of the order of microliters with highest precision.

Column

Columns used in Gas Chromatography can be either short length (1.5 – 2m) stainless steel or glass of long length (30m or above) . Capillary columns are made of fused silica flexible tubing. Packed columns are packed with stationary phase whereas capillary columns are coated with a layer of absorbing phase on the inside wall of the capillary tubing. A column is a vital component and should be maintained properly as per supplier instructions for getting long time usage and reproducible separations run after run.

Column Oven

Column Oven

Column oven houses the column and maintains constant temperature (isothermal operation) or variable temperature as per the requirement of analysis (temperature programming). The set temperature or the temperature variation should be precise and repeatable to ensure reproducibility of separation as minor temperature changes lead to changes of retention time of eluted components.

Detector

A detector provides specific response for the separated components. Majority of the organic compound applications require flame ionization detector and for specific requirements detectors such as NPD, FPD, TCD, etc. can be used.

Data Acquisition & Control

Modern GC systems are computer based and software controls operational parameters such as carrier gas flow rate, temperature programming, injection volume and sequence as well as acquisition and treatment of data output.

These are the main parts of the basic GC system configuration. Specialized applications require a combination of hardware or dedicated analyzers which are covered in our upcoming online certificate programme on GC. The subsequent Chapters will introduce you to the components in greater detail.

Role of Gases in Gas Chromatography

"Formal education will make you a living, self education will make you a fortune " — Jim Rohn

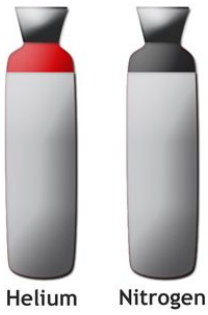
Gases play a crucial role in a Gas Chromatography system

- Transportation of injected sample to the column and subsequent transfer of separated components to the detector
- Act as fuel to support combustion in the detector
- Support combustion process in the flame when using flame ionization detection

Color coding of gases

Handling of Gases requires special precautionary measures due to their characteristics and hazard potential. Color codes have been adopted universally for easy identification and safety measures during handling.

Carrier Gas



A carrier gas facilitates transport of the injected sample to the column for separation of components of the mixture. Typical purity levels should be 99.995% or higher. Desirable features of carrier gas are :

- Inertness towards sample and stationary phase
- Should not have a response on the detector

Commonly used gases are Nitrogen, Helium and Argon. Helium gas is preferred as carrier with TCD detector because of high explosion hazard of hydrogen. Traps are recommended in the gas lines to prevent moisture or other impurities from reaching the columns.

Combustion or Fuel Gas

Hydrogen is commonly used as fuel gas when using FID detection

Hydrogen

Oxidant Gas

Zero air which is atmospheric air free of atmospheric impurities and is used to support combustion of sample in FID detector.

Zero Air

Effect of flow rate of carrier gas:

- Increase of flow rate has same effect as increase of temperature i.e. analysis speed increases but at cost of resolution
- Flow rate can be measured in terms of volumetric flow with a soap bubble flow meter, rotameter or electronic flow controller
- Mass flow meter measures rate of mass flow of gas per unit time. Mass flow rate measurements are not affected by atmospheric temperature or pressure changes and by presence of water vapour in the gas stream.

Safe Handling of gases

Gas cylinders should be placed outside the instrument room protected from direct sunlight or rain. If placed inside the instrument room they should be secured properly to a lab bench or wall with chains. While transporting from one location to another the valve should be closed and cylinder cap fixed.

Useful Tip

Use of high purity gases along with on-line traps will ensure consistency of results.

Types of Gas Chromatography Injectors

"A man is but the product of his thought, what he thinks, he becomes"

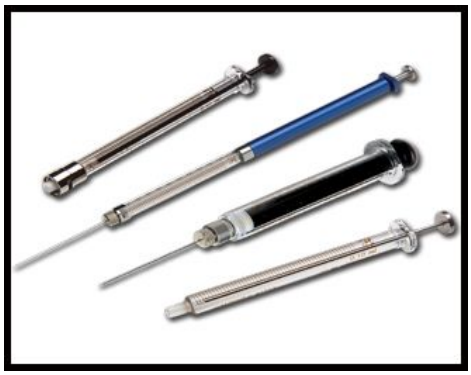
— M.K.Gandhi

Sample injection in Gas Chromatography depends on the nature of sample. Sample volume should be kept minimal for best column efficiency. The sample should enter the column as a 'plug' of vapour to avoid brand broadening and loss of resolution.

Injector

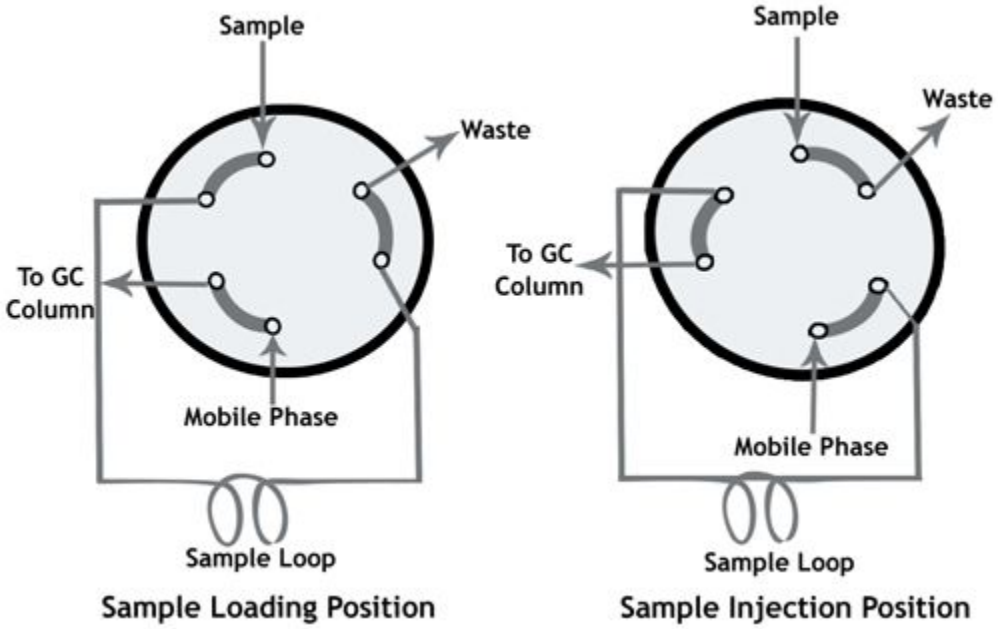
Injection of Liquid Sample

Microsyringe



Microsyringe injection is the most commonly used technique for liquid sample injection. Sample is injected through a rubber septum into a flash vaporizer. Temperature of sample port should be atleast 50OC higher than the lowest boiling component in the sample to ensure complete vaporization. Injection volumes generally range between 0.5 to 5.0 µl. Injection precision with a syringe is around ± 1%.

Injection of Gas Samples



Introduction of a constant volume of gas is more difficult with a syringe. Gas sampling valves provide high precision injection (± 0.1%). Gas sampling valves can be manually operated or automated with pneumatic or electric actuators. Gas samples in collection bottles are connected to the switching valves. The carrier gas flow is not interrupted as sample expands into a constant volume sample loop. Upon switching valve position the sample content in the loop is injected into the carrier gas stream.

Gas Chromatography Inlet Types

Column inlet is hardware attached to the column head for introduction of sample into the flowing carrier gas stream.

On-column inlet

It is a non vaporizing technique which is particularly useful for analysis of high boiling compounds like petroleum waxes, triglycerides and other thermally unstable compounds. Sample is introduced directly without heating. It enters a heated glass liner which prevents sample degradation by coming in contact with heated metal walls.

Split-splitless Injector

Split-splitless injectors are used for introduction of highly concentrated samples into capillary columns. Sample is volatilized by injection into a heated glass liner. The carrier gas then either sweeps the total sample (Splitless mode) or a portion (Split mode) into the column. The split vent controls the amount of sample entering and the other portion is exhausted. This mode is useful for highly concentrated or dirty samples. It helps in producing narrow band widths. Split-less injection is useful for trace level analysis.

Programmed Temperature Vaporizing Injection (PTV)

PTV is the technique of choice for introduction of large sample volumes (up to 250µl) to improve sensitivity. The sample is introduced into the liner at a controlled injection rate. The temperature of the liner is kept below the boiling point of the solvent. Ideal for wide boiling samples as it does not degrade thermally labile compounds.

Purge & Trap

An inert gas bubbles out volatile components in an aqueous solution. The volatiles are trapped on an adsorbent trap which on heating releases the volatiles into the carrier gas stream. Samples requiring preconcentration or purification can be introduced through such a system usually in conjunction with a split/splitless port.

Useful Tip

Flush syringe several times with sample to remove traces of past analysis samples from the needle and also clean or replace glass liner regularly.

Types of Gas Chromatography Columns

”A day spent without learning something is a day wasted”

— Anonymous

In gas chromatography, the column is the heart of the system where the separation of sample components takes place. They are classified in terms of tubing dimensions and type of packing material. Packed columns are generally 1.5 – 10m in length and 2 – 4mm id. These are generally made of stainless steel or glass. On the other hand capillary columns are 0.1 – 0.5 mm id and can be 10 – 100m long.

PACKED COLUMN FOR GAS CHROMATOGRAPHY

CAPILLARY COLUMN FOR GAS CHROMATOGRAPHY

Three types of capillary columns are commonly used in gas chromatography:

Wall Coated Open Tubular (WCOT)

Internal wall of capillary is coated with a very fine film of liquid stationary phase.

Surface Coated Open Tubular (SCOT)

Capillary tube wall is lined with a thin layer of solid support on to which liquid phase is adsorbed. The separation efficiency of SCOT columns is more than WCOT columns because of increased surface area of the stationary phase coating.

Fused Silica Open Tubular (FSOT)

Walls of capillary fused silica tubes are strengthened by a polyimide coating. These are flexible and can be wound into coils.

Column Characteristics

Column Materials

Fused silica and stainless steel columns offer high degree of inertness and flexibility. When breakage is not of much concern fused silica is the best choice.

Internal Diameter

Sample concentration is the deciding factor for the internal diameter of the column. Loss of resolution, poor reproducibility and peak distortion result if sample concentration exceeds column capacity. Typical sample loading ranges around 10ng for 0.1mm id columns to up to 2,000ng for 0.53mm id columns.

Length

Longer columns provide greater resolution of sample components. However, increasing column length increases analysis time.

Film Thickness

Film thickness determines the retention and elution temperature of each sample component. Thick films increase the time a compound stays on the stationary phase and thinner films reduce retention time. Compounds having high volatility require more residence time for better separation and should be analyzed on thicker films. The commonly used film thickness in gas chromatography columns ranges from 0.1 to 5.0µm.

Columns are selected for use in a particular application based on column length and type of packing. Guidelines on selection of columns are provided in more detail in the certificate programme which will be launched in due course.

The most important criteria in selection of column is the stationary phase packing which will be discussed in greater detail in the next Chapter.

Useful Tip

Always suspend capillary columns in stainless steel cages supplied by manufacturer to prolong the life span of the column and avoid breakages.

Types of Stationary Phases

"Optimism is the faith that leads to achievement, nothing can be done without hope and confidence" — Helen Keller

Most of the time a Gas Chromatographer is fortunate as he has to use a column specified for a particular method but there is a tendency to use the same column even though it is not the best choice for other samples. Apart from the basic column characteristics discussed in previous Chapter stationary phase choice is a critical decision for achieving the right analytical conditions.

Stationary phases are required for both Gas Solid Chromatography (GSC) and Gas Liquid Chromatography (GLC). Gas Solid Chromatography separates components in the carrier gas stream by selective adsorption on solid stationary phase whereas in GLC the components are bound to the liquid layer adsorbed on the solid support.

GLC Solid Supports

Used for separation of permanent gases and highly volatile organic compounds

Porous polymers for low carbon number organics, acids, amines and water:

- Porapak series (Millipore corp)
- Chromosorb Century series (Johns - Manville)
- Haysep (Hayes Separation)

Chromosorb – P – manufactured from hard firebrick. High load capacity.

Chromosorb – W – flux calcined diatomite.

Immobilized Liquid Stationary Phases

Desirable Features : Low volatility, High decomposition temperature and Chemical inertness.

Stationary phases are covalently bonded or cross linked liquid phases -waxes, rubbers, glasses at room temperature

Non polar Temp limit

Apiezon L <300°C
Silicone SE – 30 < 350°C
Squalene < 150°C

Desirable features of Liquid Stationary Phase

- Liquid phase should not permeate too deeply into the fine pores of the support structure as slow diffusion in and out of pores affects column efficiency
- Support should be deactivated before use as undesirable surface impurities can cause decomposition of the sample or stationary liquid layer
- Small particles of support give higher efficiency as HETP is proportional to particle diameter but particle size reduction increases back pressure.
- Liquid phase should have low volatility and high stability at elevated temperatures otherwise they can contribute to interference in analysis.

Typical Liquid Phases

Stationary Phase	Trade Name	Max Temp	Common Applications
Dimethyl Polysiloxane	OV – 1, SE – 30	350°C	Hydrocarbons, Polynuclear aromatics, PCB's
Poly(phenyl methyl) siloxane	OV – 17	250°C	Steroids, Pesticides, Glycols
Poly (Trifluoro propyl dimethyl) siloxane	OV – 210	200°C	Chlorinated Aromatics, Nitro Aromatics, Alkyl substituted Benzenes
Polyethylene Glycol	Carbowax 20	250°C	Free acids, Alcohols, Essential Oils,

	M		Glycols
5% Diphenyl – 95% Dimethyl polysiloxane	DB – 5	325°C	Flavors, environmental samples and aromatic hydrocarbons

Chiral Stationary Phases

Chairal stationary phases separate optical isomers by selective interactions such as hydrogen bonding metal bond coordination and also in cooperation of modified cyclodextrins. The chiral selector is anchored to a polycyloxane back bone. GC column coated with modified cyclodextriens are applied for enantiomer analysis in flavors, essential oils, terpenoids, pheromones, enzymatic reactions and organo chloride pesticides.

Types of Gas Chromatography Detectors

"If you want to increase your success rate, double your failure rate"

— Thomas Watson, Sr. founder of IBM

The Gas Chromatography detector is capable of establishing both the identity and concentration of eluting components in the carrier gas stream. Before going further into the types of detectors it is essential to understand the nature of detectors and their desirable characteristics.

- **Non-selective**

o Responds to all compounds present in carrier gas stream except the carrier gas itself

- **Selective**

o Responds to range of compounds with a common physical or chemical characteristic

- **Specific**

o Responds to a single specific compound only

Detectors can also be grouped into concentration or mass flow detectors

Concentration Dependent

The response of such Gas Chromatography detectors is proportional to the concentration of the solute in the detector such as TCD. Dilution of sample with makeup gas will lower detector response.

Mass Flow Dependent

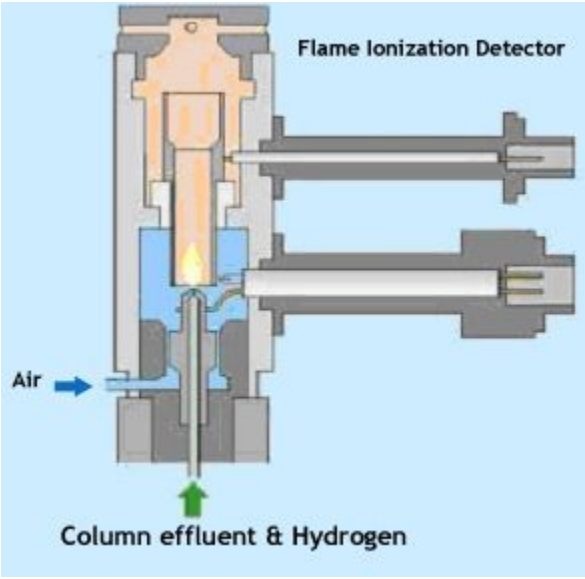
Signal is dependent on the rate at which solute molecules enter the detector such as FID. Response of such detectors is not affected by makeup gas flow rate changes.

Desirable characteristics of detectors :

- Reproducible response to changes in eluent composition in carrier gas stream
- High sensitivity
- Large linear dynamic range
- Low noise
- Small volume to avoid peak broadening and resultant loss of resolution
- Preferably non – destructive

Common Gas Chromatography detectors

Flame Ionization Detector (FID)



- Mass sensitive detector
- Response depends on conducting power of ions or electrons produced on burning of organic compounds in the flame

- Selective detector but sample detected must be combustible
- Large linear dynamic range (10^7)
- No response to inorganic and permanent gases such as CO, CO_2, NH_3, CS_2, N_2, etc.
- It is the most widely used detector in Gas Chromatography

Thermal Conductivity Detector (TCD)

- Non-destructive universal detector
- Response depends on the thermal conductivity difference between the carrier gas and the eluted components
- Wide dynamic range (10^7 - % to ppm levels)
- Responds also to inorganic gases such as CO, CO_2, NH_3, CS_2, N_2, etc.

Electron Capture Detector (ECD)

- Operation based on absorption of β – rays emitted by radioactive source Ni63. Electrophores absorb the β – rays thereby reducing the current in the detector
- Specific detector, non-destructive in nature
- Linear dynamic range about 10^5
- Widely used in environmental analysis e.g. organochlorine pesticide

Nitrogen Phosphorous detector (NPD)

- Ions migrate to the collector electron creating a current proportional to sample concentration

- Responds selectively to most organic compounds that contain phosphorus or nitrogen down to picogram levels
- Mass flow dependent detector
- Linear range about 10^6
- Useful for analysis of drugs and pesticides containing phosphorus

Gas Chromatography Applications

"Success doesn't come to you, you go to it" — Marva Collins

Gas Chromatography has been applied to identify and quantify the components of thousands of compounds present in complex matrices covering pharmaceuticals, foods & beverages, environmental samples, petroleum, polymers, forensic Science, etc. The application is further extended by choice of columns, system Chapters, detectors and use of hyphenated techniques such as GC – MS, GC - FT- IR, etc.

The major application areas are outlined in this Chapter.

Pharmaceuticals

- Residual solvents in intermediate and finished products
- Related substances and volatile impurities
- Drug assays

Foods and Beverages

- Residual pesticide, herbicides etc.

- Food adulteration
- Analysis of Fragrances and flavors
- Fatty acids profiling of fats and oils
- Fatty Acids Methyl Esters (FAME) analysis

Environmental Analysis

- Ambient air and stack emissions monitoring
- Analysis of Volatile components in waste water
- Residual pesticide in agricultural produce, vegetables, fruits, soils and drinking water
- Analysis of dioxins, polynuclear aromatic hydrocarbons, polychlorinated biphenyls (PCB's), dibenzofurans, etc.

Petroleum

- Petroleum distillates
- Refinery gases
- Analysis of LPG, compressed natural gas, petroleum ether, light naptha, gas oil, lubricating oil and gases in lubricating oils

Modified analyzers such as :

- Refinery Gas Analyzers (RGA)
- Natural Gas Analyzers (NGA)
- Paraffins, Isoparaffins, Olefins, Napthalenes and Aromatics Analyzers (PIONA/ PONA)
- Stimulated Distillation Analyzers (SMDIS)

The application scope of gas chromatography is unlimited. More applications are discussed in the certificate programme which will be launched in due course.

We are sure you have gained exposure to basics and typical applications of gas chromatographic technique. The concluding Chapter will cover 10 frequently asked questions on GC technique that you may encounter in job interviews.

Top 10 Interview Questions on Gas Chromatography

"Education costs money, but then so does ignorance" — Claus Moser

Your understanding of a topic and technical skills are gauged in interviews. We now provide you an opportunity to familiarize yourself with some typical questions that you may face in interviews when seeking career opportunities in analytical laboratories involving extensive use of gas chromatography technique.

Q1. What are the main differences between High Performance Liquid Chromatography and Gas Chromatography?

Ans. In HPLC the mobile phase is a liquid whereas in Gas Chromatography the mobile phase or carrier is a gas.

- HPLC is useful for analysis of samples which are liable to decompose at higher temperatures. GC involves high temperatures so compounds are stable at such temperatures.
- Gas Chromatography is applied for analysis of volatile compounds whereas non volatile compounds can be easily analyzed on HPLC
- Gas Chromatography cannot be used for analysis of high molecular weight molecules whereas HPLC has applications for separation and identification of very high molecular weight compounds
- HPLC requires higher operating pressures than GC because liquids require higher pressures than gases for transport through the system
- HPLC columns are short and wide in comparison to GC columns

Q2. Which type of GC detector is most commonly used? Explain its working principle and what are its limitations?

Ans. The most commonly used detector is the flame ionize detector. The sample is combusted with the help of fuel gas and oxidant in the detector body. Combustible sample components burn and produce ions and electrons which can conduct electricity through the flame. A large potential difference is applied at the burner tip and the collector electrode located above the flame and the current between the electrodes is measured. The detector is mass sensitive and response is not affected by carrier gas flow rate changes. However, the detector is not responsive to inorganic gases such as CO, O2, NH3, N2, CS2, CO2, etc.

Q3. What are the commonly used carrier gases in GC analysis when using FID detector?

Ans. Inert gases commonly used in analysis when using FID detector are Nitrogen and Helium. Nitrogen is more commonly used as it is less expensive than Helium. Purity of carrier gas should be more than 99.995% and on-line traps should be used to prevent residual moisture or other impurities from entering the system.

Q4. What are the desirable characteristics of a GC detector ?

Ans. The detector chosen for particular analysis should :

- Give reproducible response to changes in concentration of eluting compounds in the carrier gas stream.
- Should provide a large linear dynamic range
- Should have high sensitivity
- Should have small internal volume to give narrow peaks and also facilitate flushing of previous sample traces
- Should preferably be non-destructive

Q5. What do you understand by specificity of a detector?

Ans. Detectors falls into three categories depending upon response to the eluting compounds:

- Non-selective – Respond to all component in the gas stream except for the carrier gas
- Selective – Respond to a particular class of compounds with common physical or chemical properties
- Specific – Respond to a single specific compound only in the carrier gas stream

Q6. What are the commonly used types of capillary columns?

Ans. Capillary columns are generally 10 – 100m long tubes having an internal diameter ranging from 0.1 – 0.5mm made of flexible material such as fused silica. Common types of capillary columns are

- Wall coated open tubular (WCOT) – Internal wall is coated with a very fine film of adsorbing liquid
- Surface coated open tubular (SCOT) – Inner wall is lined with a layer of solid support on to which the liquid phase is absorbed.

The columns are flexible and wound into several turn coils supported on a SS cage inside the column oven

Q7. What do you understand by column efficiency and how it is expressed?

Ans. On continuous use a column gradually loses its original resolution power. Column efficiency is expressed on the basis of plate theory concept. Each component under separation spends a finite time in each theoretical plate. Smaller the plate height the larger the number of plates (N) and better is the column efficiency.

$N = 16 (t_r/w)^2$

where N = Number of theoretical plate
tr = Retention time of a peak

w = Peak width at base

N can also be expressed as

$N = 5.54(t_r/W_{1/2})^2$

where $w_{1/2}$ is peak width at half peak height

Column efficiency is expressed in terms of Height Equivalent to a Theoretical Plate (HETP)

HETP = L/N

where, L = Length of column in terms of mm

Small value of HETP signifies greater column efficiency

Q8. What do you understand by temperature programming in GC analysis?

Ans. Temperature programming means change of temperature of the column at a rate predetermined rate during the analytical run. This has the same influence on elution time of separated components as gradient programming in HPLC analysis. Temperature programming helps reduce analysis time by permitting early elution of less volatile components.

Q9. When is isothermal operation useful?

Ans. Isothermal operation is useful when high resolution is required for separating compounds having narrow boiling range. Temperature is set to around mid range of boiling points of constituents. This results in good resolution of low boiling components but band broadening of higher boiling components can result due to their longer retention in the column.

Q10. What measures you would adopt to extend useful life of a column?

Ans.

- Condition a column before first use or after long time storage
- Take care not to exceed upper temperature limit specified by the manufacturer
- Avoid injection of solutions which are strongly acidic or basic in nature

- Rinse columns by injection with blank solvents such as methanol, methylene chloride or hexane to remove contamination of column after excessive usage

Conclusion

We believe that you enjoyed the free e-book on GC. The course provided you an insight into the components of GC system and their individual contribution towards the overall accuracy and precision of your results. Apart from a general introduction the course was designed keeping in mind the requirements of the GC user. Without going into mathematical treatment of the subject an attempt has been made to convey the basics concepts and offer practical tips on effective utilization of the GC system.

The last chapter of the course provides answers to 10 common questions that you may be faced with as you move up your career ladder. However, learning is a lifelong process and there will be several unanswered questions and queries which will be coming up in your mind from time to time. Our suggestion to you would be to post such queries or comments on the site and we shall try to offer clarifications to the best of our ability based on our expertise and experience.

In case the e- course has awakened your desire to go deeper into the subject you are welcome to join the Certificate Course on GC which would be available round the year after its launch.. For further details on this advanced treatment of the subject please await our announcements on the site.

Once again we take the opportunity to thank you for your interest. Please feel free to participate actively by contributing articles in areas off your interest and offer your valuable comments and suggestions